



Synthesis and Characterization of Poly Ortho Toluidine Doped with Commercial TiO₂ Nanoparticle

V. Gayathri¹, R. Balan^{2*}

^{1,*2}Department of Physics, Chikkanna Government Arts College, Trippur, TN, India.

Received: 21.08.2019 Accepted: 25.09.2019

Abstract

The aim of this work was to study the changed properties of commercial TiO₂ with conducting polymer such as poly ortho toluidine were synthesized by chemical oxidative polymerization. The metastable anatase phase of TiO₂ and POT+ TiO₂ composites were characterized by using X-Ray Diffraction analysis (XRD), Scanning Electron Microscopy (SEM-EDX) and Fourier Infrared Spectroscopy (FTIR). The bandgap of samples find out by UV-Visible spectroscopy.

Keywords: Commercial TiO₂, FT-IR, Polyortho toluidine (POT), SRD, UV-Vis.

1. INTRODUCTION

Conducting polymer such as poly aniline is considered to be unique due to its excellent electrical and optical properties. Poly aniline have been used in variety of applications such as corrosion protection, catalysis, sensors because of its non-redox doping, good environmental and thermal stability, high conductivity and economic feasibility. The properties of conducting polymer composites depends upon the doping concentration of nanoparticles (Tieli Zhou *et al.* 2015; Kiran Kumari *et al.* 2011). Among conducting polymers poly ortho toluidine is highly attracted and it has better solubility and higher processability by steric hinderence π -electron effects (Gigliola Lusvardi *et al.* 2017). TiO₂ is a semi conducting nanoparticles play an important role in wide range of applications due to its good photo catalytic activity and used for large scale production, cosmetics and air purification. The three types of crystallographic structures of TiO₂ are anatase, rutile and brookite. Among all these phases rutile is the most stable one where anatase and brookite corresponds to metastable and unstable states (Bin Wang *et al.* 2015).

The aim of this work was to study the changed properties of TiO₂ due to the presence of poly ortho toluidine in the composites. Chemical oxidative polymerization method is used to synthesize poly ortho toluidine. Here we report the synthesis as well as structural and optical properties of poly ortho toluidine

doped with TiO₂ nanoparticles. The functional groups of materials were determined by Fourier infra red spectroscopy (FTIR). The functional groups of samples were examined by FTIR spectroscopy. The morphology, structural and band gap of TiO₂ and Poly ortho toluidine/TiO₂ composites can be carried out by SEM-EDX, XRD and UV spectroscopy.

2. EXPERIMENTAL DETAILS

2.1 Materials & method

O-toluidine (99% AR grade, Lobal chemie laboratory reagents & fine chemicals), H₂SO₄, Ammonium peroxy disulfate (>98%, Merck), Commercial TiO₂ (98%, Fisher scientific), Millipore – Q water were used as received.

Poly ortho toluidine- TiO₂ composites has been synthesized by chemical oxidative polymerisation at room temperature. In this process, an appropriate amount of size reduced TiO₂ is dispersed in 0.2 M of H₂SO₄ by ultrasonication. After that this dispersed TiO₂ solution is added slowly to 0.1 M aqueous solution of O-toluidine with continuous stirring for 30 minutes. 0.1 M aqueous solution of ammonium peroxy disulfate was added dropwise to polymerisation bath to initiate the polymerization and the solution was left for 4 hr with continuous stirring. Later the solution was allowed to kept for overnight. The resultant precipitate was filtered and washed several times with acetone followed by

* R. Balan

email:balanphy@yahoo.co.in

water and to be dried at 80 °C in an oven. Finally dark green colour powder form of POT/ TiO₂ composite can be obtained.

3. MATERIAL CHARACTERIZATION

X-ray diffraction data of samples were recorded using XPERT diffractometer. The surface morphology and elemental compositions have been investigated by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Absorption spectra of anatase phase of TiO₂ and composites were recorded by using UV-Visible spectrometer (DRS), Analytek Jena, Germany. The functional groups of samples were found by ATR-FTIR, Bruker, Germany.

4. RESULTS & DISCUSSION

4.1 X-ray diffraction analysis

The XRD characteristic peaks at 25°, 31°, 37° in fig.1a represents the anatase phase of TiO₂

corresponding to the (101) (004) (200) planes. The XRD spectra of POT+ TiO₂ in fig.1b is similar to that of pure TiO₂ shows that the crystalline phase of TiO₂ did not change by poly ortho toluidine. The only difference is that the intensity of diffraction peaks of POT+ TiO₂ composites was slightly lesser than that of pure TiO₂ due to the amorphous nature of POT in the composites.

4.2 Morphological analysis

The morphology of pure TiO₂ and POT+ TiO₂ were examined by scanning electron microscopy. Fig.2 (a) shows the SEM micrographs of TiO₂ nanoparticles are spherical in shape where fig.2 (b) reveals that flaky like feature with globular structure of POT interlinked with TiO₂ nanoparticles which confirm the formation of composites. Fig.3 (a & b) shows the elements corresponds to TiO₂ and POT+ TiO₂ composites.

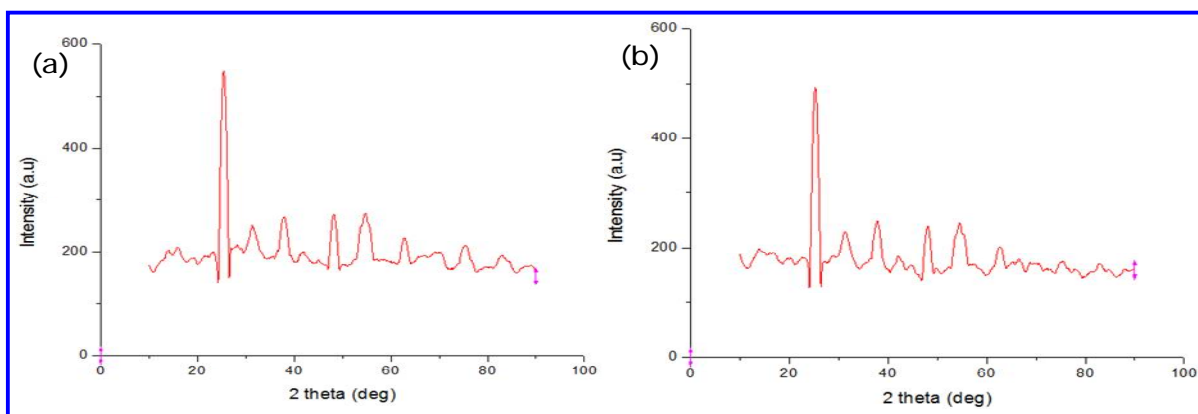


Fig. 1: XRD of (a) TiO₂ nanoparticle (b) POT + TiO₂ composites

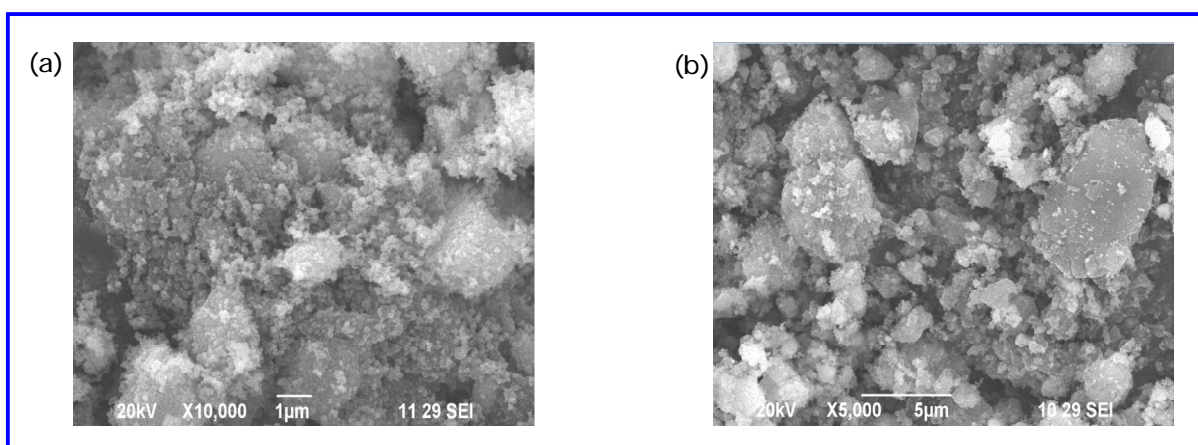


Fig. 2: SEM images of (a) TiO₂ nanoparticle (b) POT + TiO₂ composites

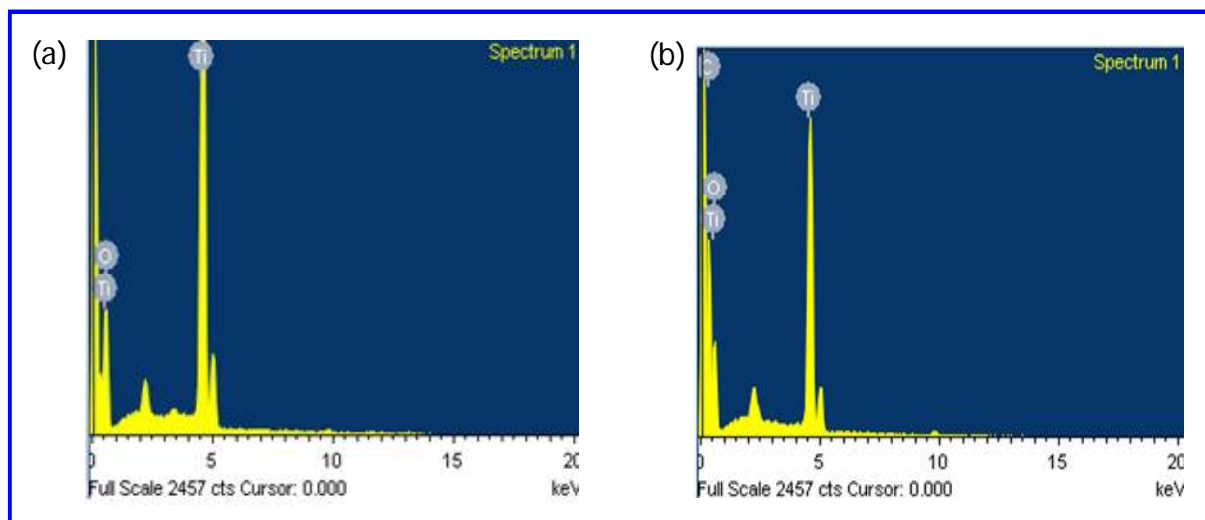


Fig. 3: EDX spectra of (a) TiO_2 nanoparticle (b) POT + TiO_2 composites

4.3 UV Vis Spectroscopy

In fig.4 (a & b) the absorbance of TiO_2 and POT+ TiO_2 composites is about 344 nm and 334 nm respectively. The absorbance of composites is slightly shifted to lower wavelength indicating the dispersion of TiO_2 in the composites. The band gap of pure TiO_2 and POT+ TiO_2 can be calculated by using the formula $E=h\nu$. The band gap value of composites is 3.7 eV which is greater than pure TiO_2 (3.6 eV).

4.4 FOURIER INFRA RED SPECTROSCOPY

In Fig.5 (a & b) shows the FTIR spectra of pure TiO_2 and POT+ TiO_2 composites. It can be seen from fig.3(a), the characteristic peaks at 3749 cm^{-1} and 604 cm^{-1} indicate the Ti-O-Ti and O-H stretching frequencies (Asha *et al.* 2014). The intensity of characteristic peaks corresponds to composites is somewhat similar to that of pure TiO_2 with additional peaks at 3619 cm^{-1} corresponds to the N-H stretching of an aromatic amine and it confirms the formation of POT in the composites (Borriello *et al.* 2011). The peaks observed at 1494 cm^{-1} in composites corresponds to C-N stretching vibrations of quinoid and benzenoid rings (Canon Uchoyuk *et al.* 2012).

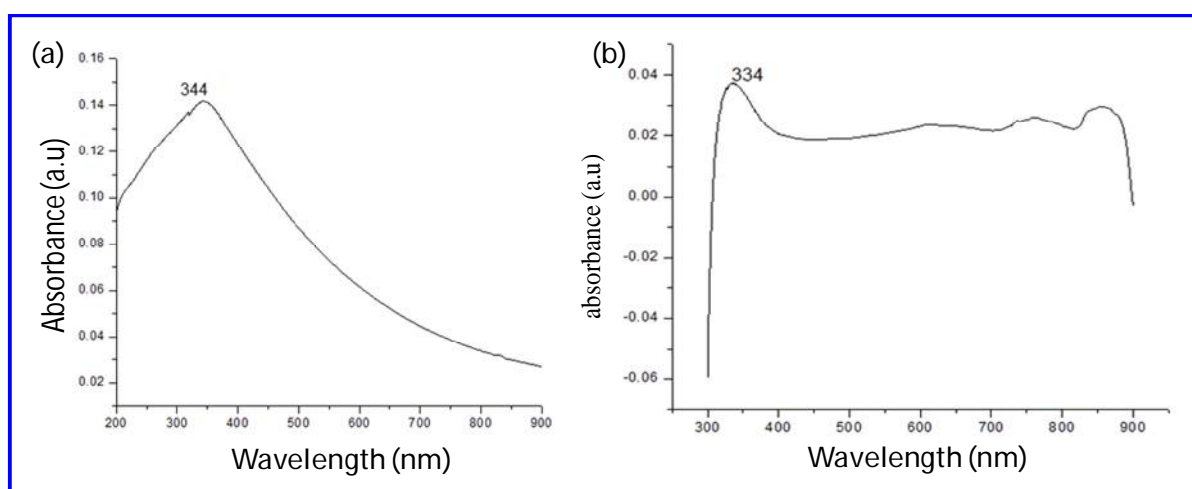


Fig. 4: UV –Visible spectra of (a) TiO_2 nanoparticle (b) POT + TiO_2 composites

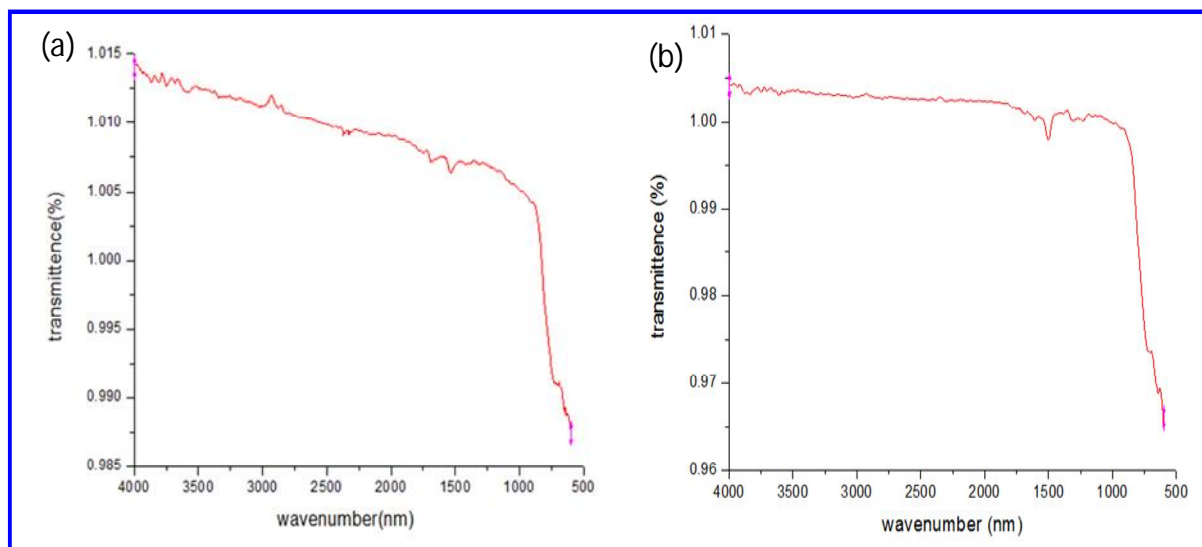


Fig. 5: FT-IR spectra of (a) TiO₂ nanoparticle (b) POT + TiO₂ composites

5. CONCLUSION

The changed properties of TiO₂ due to the presence of poly ortho toluidine were synthesized by chemical oxidative polymerization method. XRD studies show that the crystalline structure of anatase TiO₂ with amorphous nature of poly ortho toluidine, which confirms the formation of composites with lesser intensity. The functional groups and spherical morphology of TiO₂ interlinked with poly ortho toluidine was examined by FT-IR and SEM-EDX. UV-Visible spectroscopic results show that POT+ TiO₂ composites exhibit higher band gap when compared to pure TiO₂.

REFERENCES

- Asha, Sneha Lata Goyal, Kumar. D, Shyam Kumar and Kishore.Nn., Synthesis and characterization of polyaniline/TiO₂ composites, Indian Journal of Pure and Applied Physics, 52(2), 341-347 (2014).
- Bin Wang, Hongji Qi, Hu wang, Yanyan Cui, Jiaoling Zhao, Jialu Guo, Yun Cui, Yuchen Liu, Kui Yi and Jiando Shao, Morphology, structure and optical properties in TiO₂ nanostructured films annealed at various temperatures, Optical Materials Express, 5(6), 1410-1418 (2015). [doi:10.1364/OME.5.001410](https://doi.org/10.1364/OME.5.001410)
- Borriello, A., Guarino, V., Schiavo, L., Alvarez Perez, M. A. and Ambrosio, L., Optimizing PANi doped electroactive substrates as patches for the regeneration of cardiac muscle, J. Mater. Sci. Mater. Med., 22(4), 1053-1062(2011). [doi:10.1007/s10856-011-4259-x](https://doi.org/10.1007/s10856-011-4259-x)
- Canon Uchoyuk, Meral Karakislal and Mehmet Sacak., Preparation of Poly (o-toluidine) / Polyacrylonitrile composite fibers using CrO₃, Indian Journal of Fiber and Textile Research, 37(2), 120-126(2012).
- Gigliola Lusvardi, Corrado Barani, Federica Giubertoni, Giulia paganelli.: Synthesis and characterization of TiO₂ nanoparticles for the reduction of water pollutants, Materials, 10(10), 1208-1218(2017). [doi:10.3390/ma10101208](https://doi.org/10.3390/ma10101208)
- Kiran Kumari, Vazid Ali, Gita Rani, Sushil Kumar, Lakshmi, G. V. B. S. and Zolfequar, M., DC conductivity and spectroscopic characterization of poly (o-toluidine) doped with binary dopant ZrOCl₂/AgI, Materials Sciences and Application, 2(8), 1049-1057(2011). [doi:10.4236/msa.2011.28142](https://doi.org/10.4236/msa.2011.28142)
- Tieli Zhou, Xiuhong Xie, Jianyan Cai, Liying Yin and Weidong Ruan, Preparation of Poly (o-toluidine) / TiO₂ nanocomposite films and application for humidity sensing, Polym.Bull., 73(3), 621-630(2015). [doi:10.1007/s00289-015-1509-y](https://doi.org/10.1007/s00289-015-1509-y)